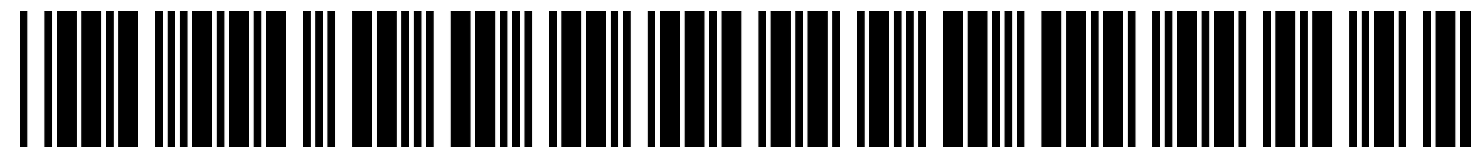


US007798001B2

(12) **United States Patent**
Shekhawat et al.

(10) **Patent No.: US 7,798,001 B2**
(45) **Date of Patent: *Sep. 21, 2010**

(54) **SCANNING NEAR FIELD ULTRASOUND HOLOGRAPHY**

(75) Inventors: **Gajendra Shekhawat**, Arlington Heights, IL (US); **Vinayak P. Dravid**, Glenview, IL (US)

(73) Assignee: **Northwestern University**, Evanston, IL (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

This patent is subject to a terminal disclaimer.

(21) Appl. No.: **12/244,406**

(22) Filed: **Oct. 2, 2008**

(65) **Prior Publication Data**

US 2009/0114024 A1 May 7, 2009

Related U.S. Application Data

(63) Continuation of application No. 11/244,747, filed on Oct. 6, 2005, now Pat. No. 7,448,269, which is a continuation-in-part of application No. 10/913,086, filed on Aug. 6, 2004, now abandoned.

(60) Provisional application No. 60/494,532, filed on Aug. 12, 2003.

(51) **Int. Cl.**
G01N 29/04 (2006.01)
G01V 1/00 (2006.01)

(52) **U.S. Cl.** **73/603**; 73/596; 181/101

(58) **Field of Classification Search** 73/603, 73/105, 596, 584; 250/306, 307; 181/101; 331/15, 23, 41, 42, 107
See application file for complete search history.

(56) **References Cited**

U.S. PATENT DOCUMENTS

3,617,754 A * 11/1971 Hildebrand 250/216
3,889,226 A * 6/1975 Hildebrand 367/8
4,170,142 A 10/1979 Posakony et al.
4,222,273 A * 9/1980 Takahashi et al. 73/603
4,871,984 A 10/1989 Laton et al.
5,410,742 A 4/1995 Yajima
5,503,010 A * 4/1996 Yamanaka 73/105
5,675,075 A 10/1997 Arnold et al.
5,804,708 A * 9/1998 Yamanaka et al. 73/105
5,821,410 A * 10/1998 Xiang et al. 73/105

(Continued)

FOREIGN PATENT DOCUMENTS

WO WO 98/08046 2/1998

OTHER PUBLICATIONS

Cuberes et al., Heterodyne Force Microscopy: Nanomapping of Viscoelastic Response at Ultrasonic Speeds, Abstract of STM '99 conference, Jul. 1999, Seoul, Korea.

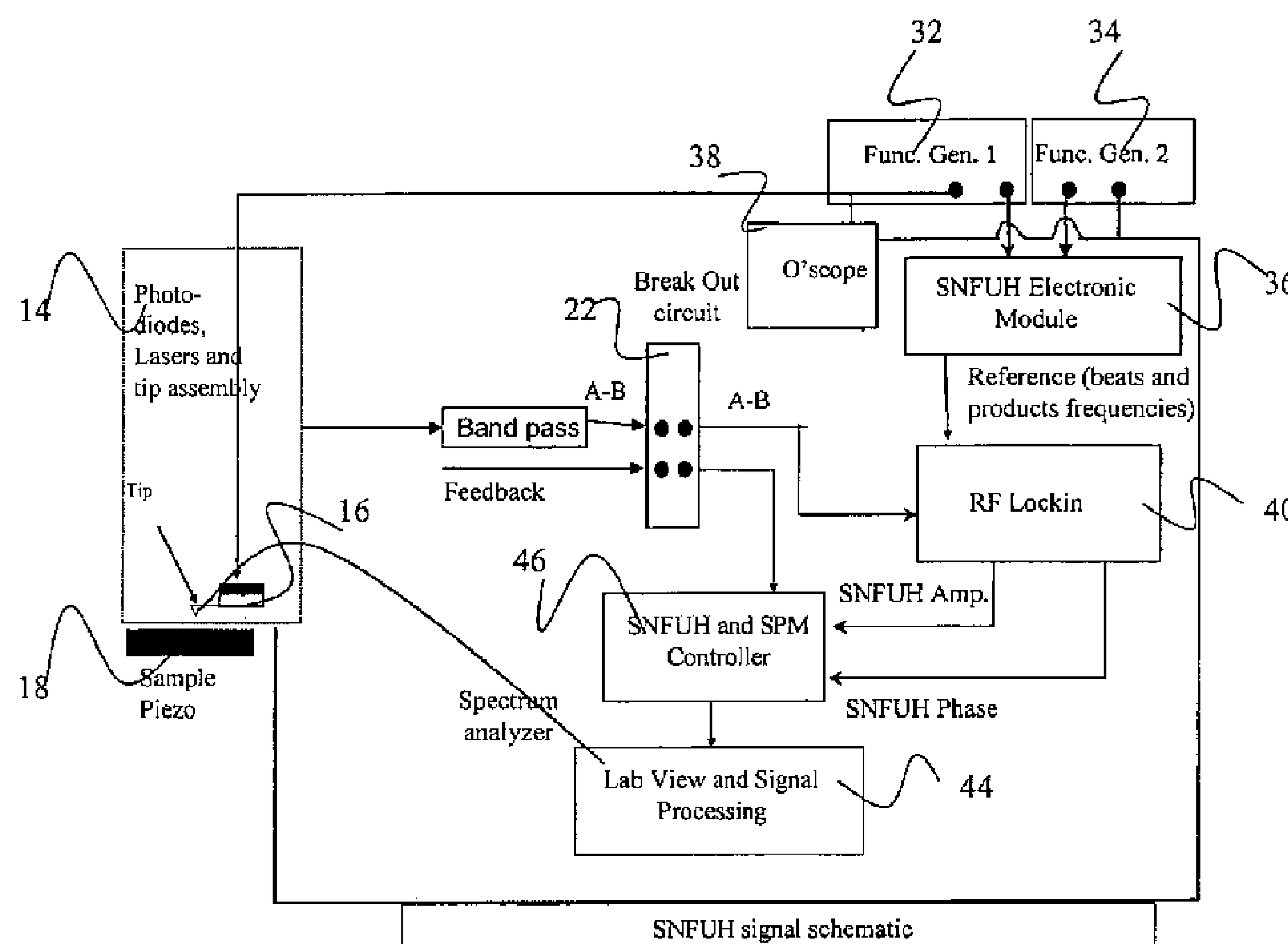
(Continued)

Primary Examiner—J M Saint Surin
(74) *Attorney, Agent, or Firm*—McAndrews, Held & Malloy, Ltd.

(57) **ABSTRACT**

A high spatial resolution phase-sensitive technique employs a scanning near field ultrasound holography (SNFUH) methodology for imaging elastic as well as viscoelastic variations across a sample surface. SNFUH uses a near-field approach to measure time-resolved variations in ultrasonic oscillations at a sample surface. As such, it overcomes the spatial resolution limitations of conventional phase-resolved acoustic microscopy (i.e. holography) by eliminating the need for far-field acoustic lenses.

20 Claims, 9 Drawing Sheets



U.S. PATENT DOCUMENTS

5,840,041	A *	11/1998	Petter et al.	600/547
6,006,593	A *	12/1999	Yamanaka	73/105
6,049,728	A	4/2000	Chou	
6,094,072	A *	7/2000	Davies et al.	326/98
6,094,972	A	8/2000	Yasutake et al.	
6,133,795	A	10/2000	Williams	
6,353,576	B1	3/2002	Garlick et al.	
6,708,556	B1	3/2004	Kim et al.	
6,798,796	B2	9/2004	Hiroshi et al.	
6,831,874	B2	12/2004	Garlick et al.	
6,876,791	B2	4/2005	Khoury	
6,943,924	B2	9/2005	Marquet et al.	
6,666,075	B2	12/2005	Mancevski et al.	
6,983,644	B2	1/2006	Yamanaka et al.	
7,360,404	B2 *	4/2008	Reinstadtler et al.	73/105
7,448,269	B2 *	11/2008	Shekhawat et al.	73/603
7,615,738	B2 *	11/2009	Kley	250/234
2002/0057757	A1	5/2002	Khoury	
2003/0152393	A1	8/2003	Khoury	
2007/0220978	A1 *	9/2007	Su et al.	73/632

OTHER PUBLICATIONS

Kolosov et al., Ultrasonic Force Microscopies - merging ultrasound and SPM to explore nanometre scale mechanics on the nanosecond time scale, Extended Abstract of STM '99 conference, Jul. 1999, Seoul, Korea.

Cuberes et al., Heterodyne force microscopy of PMMA/rubber nanocomposites: nanomapping of viscoelastic response at ultrasonic frequencies, J. Phys. D: Appl. Phys. 33, 2000, pp. 2347-2355, IOP Publishing Ltd.

Geer et al., Nanometer-scale mechanical imaging of aluminum damascene interconnect structures in a lowdielectric-constant polymer, Journal of Applied Physics, Apr. 1, 2002, pp. 4549-4555, vol. 91, No. 7.

Kolosov, UFM shakes out the details at the nanoscopic scale, Materials World, Dec. 1998, pp. 753-754.

* cited by examiner

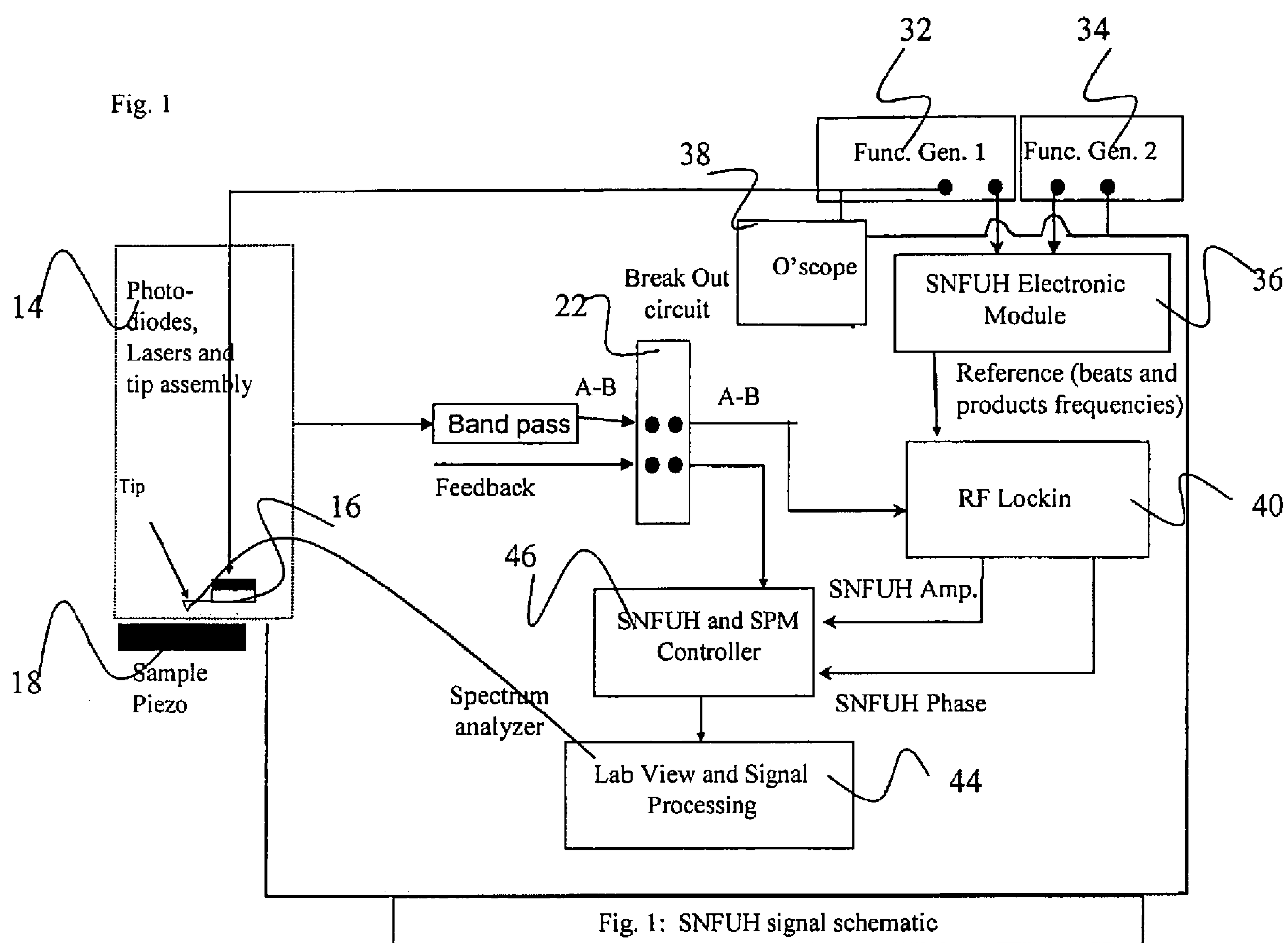


Fig. 1: SNFUH signal schematic

Fig. 2

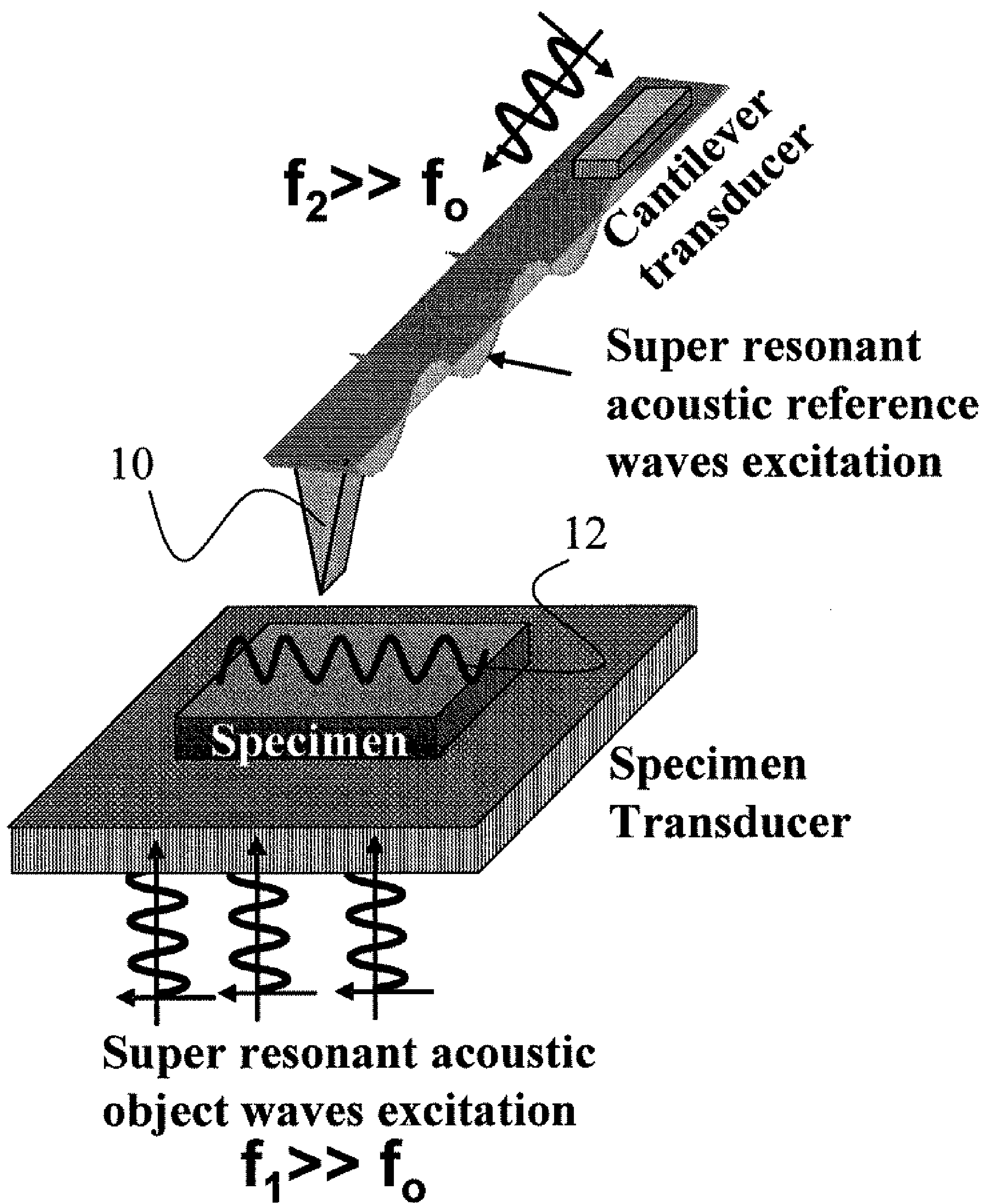


Fig. 3

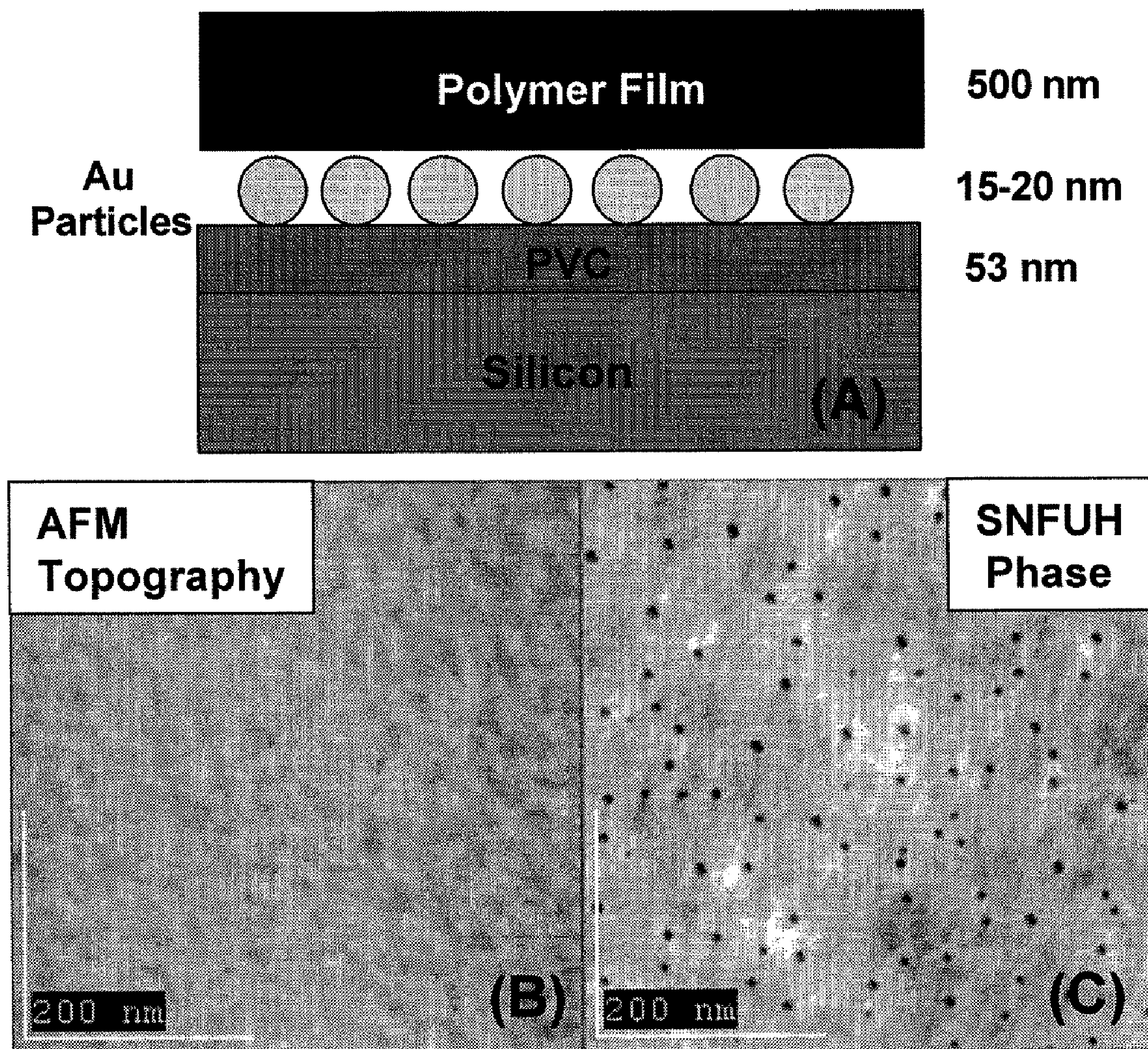


Fig. 4

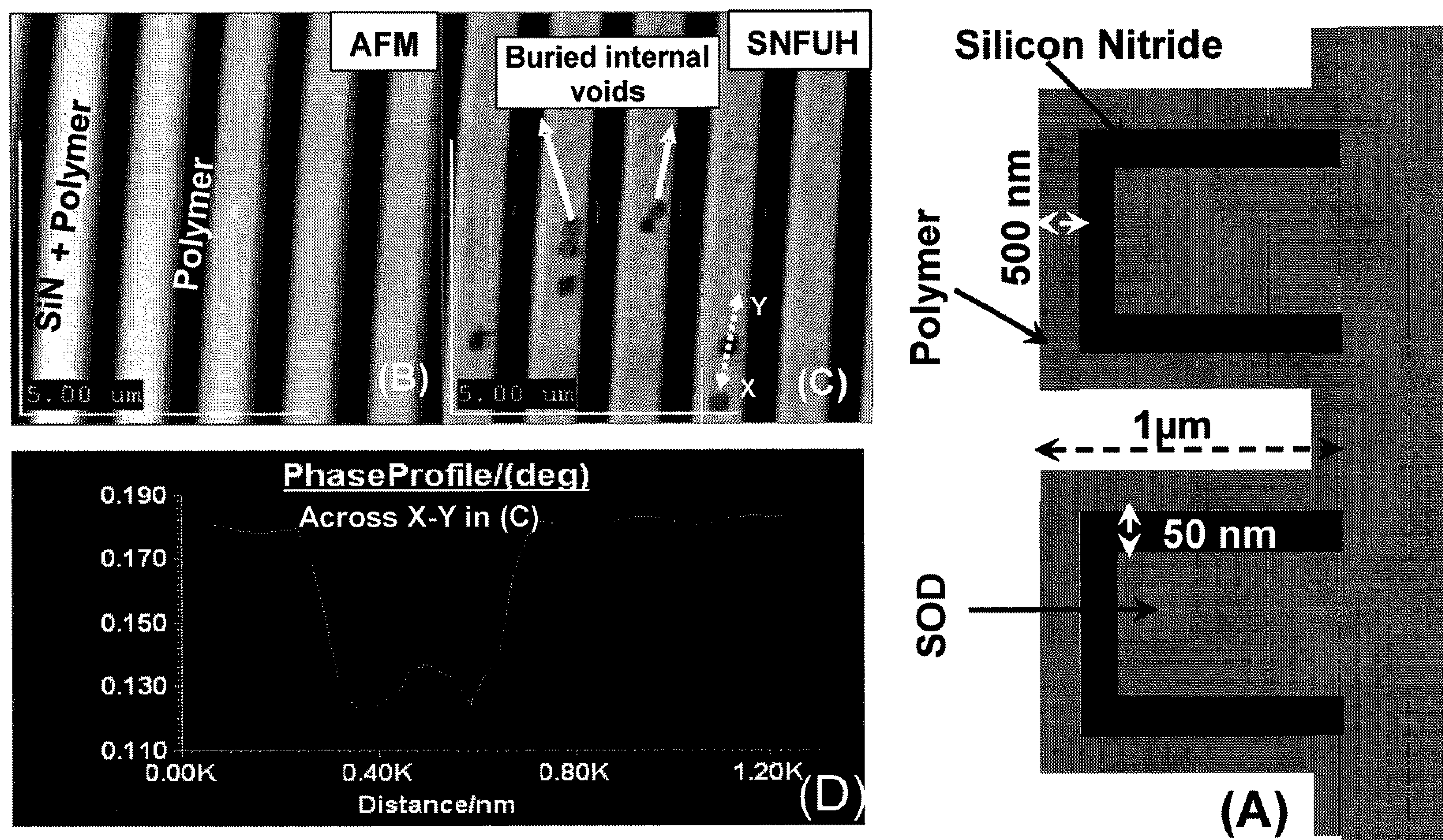


Fig. 5

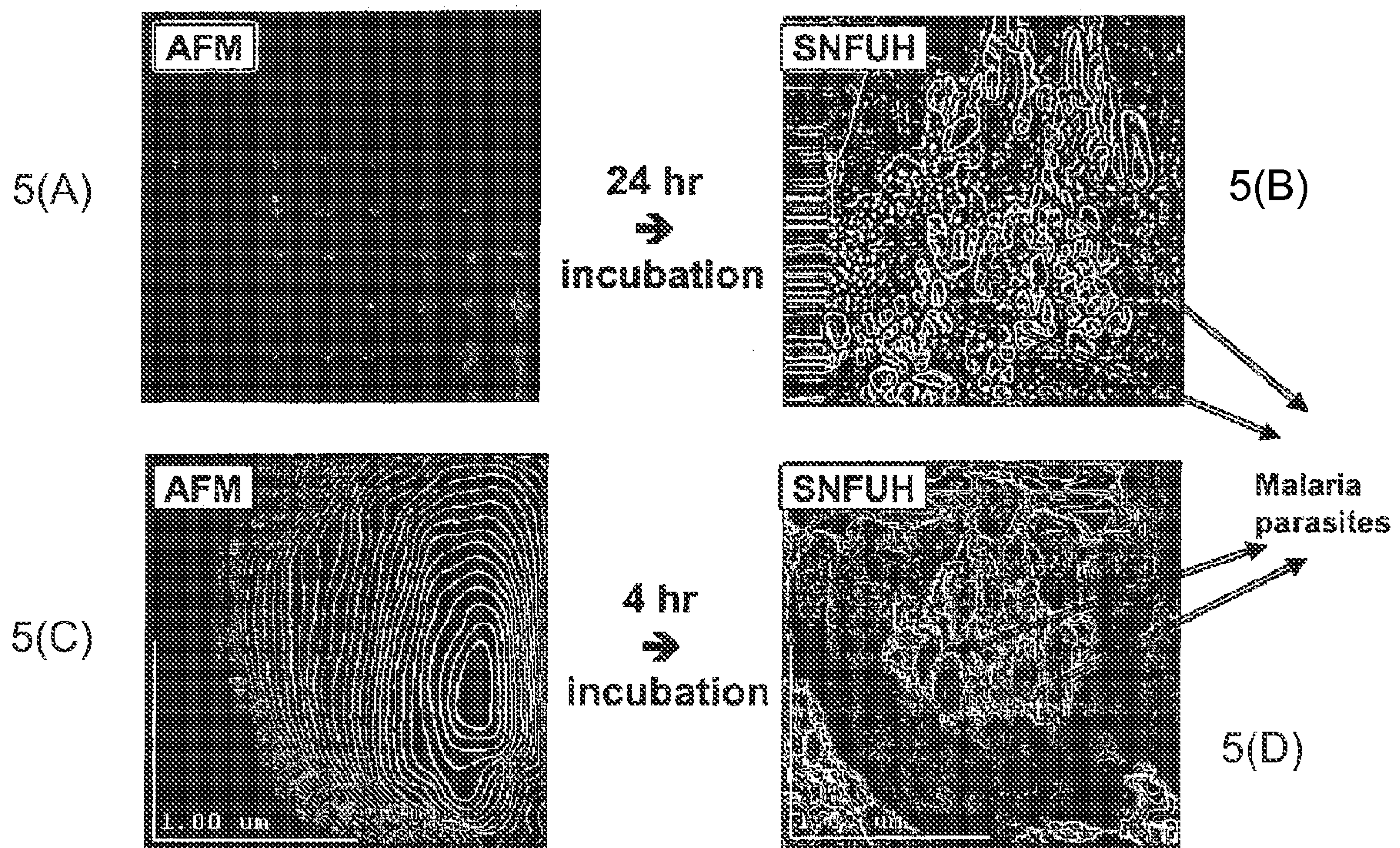
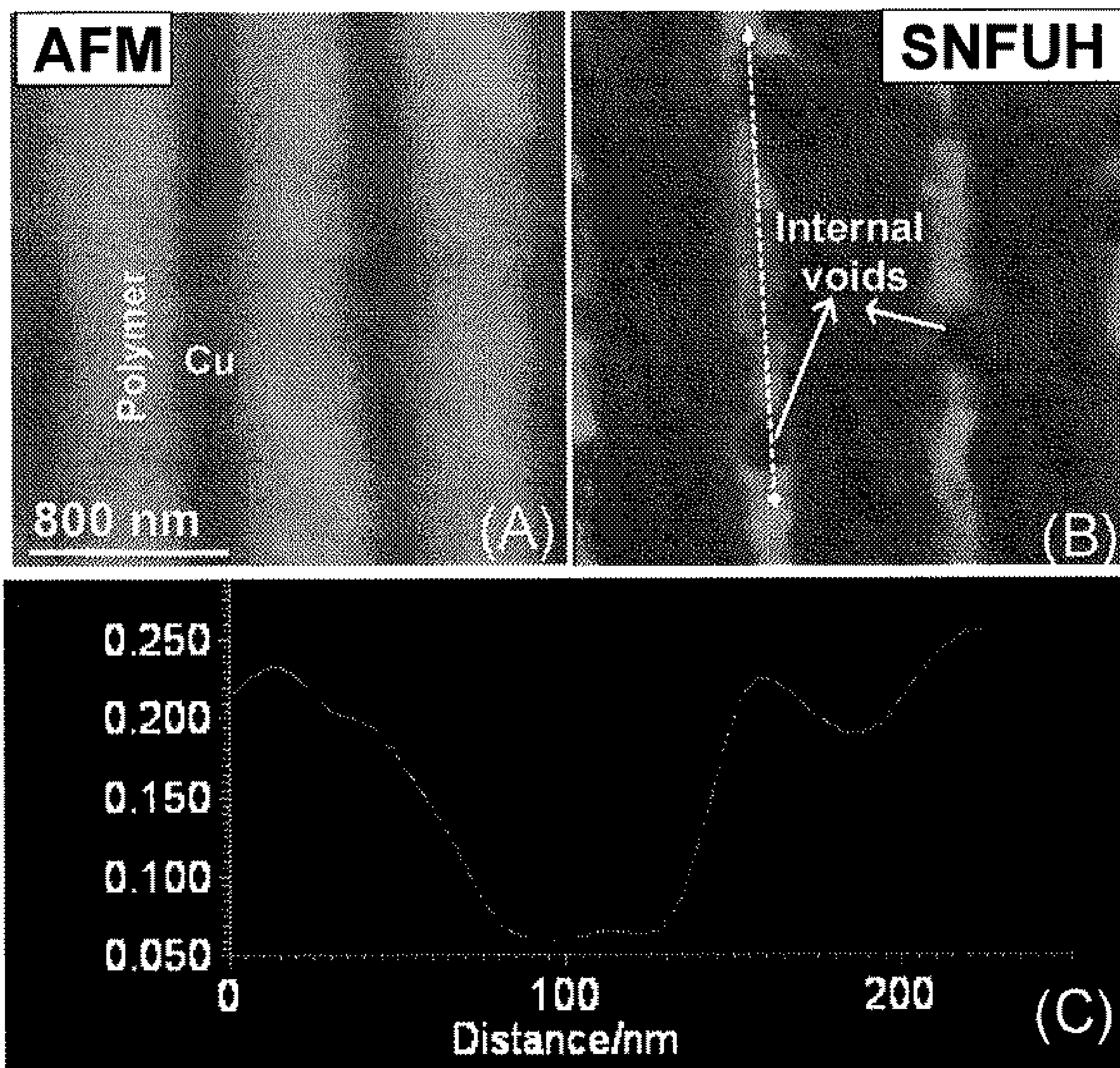


Fig. 6



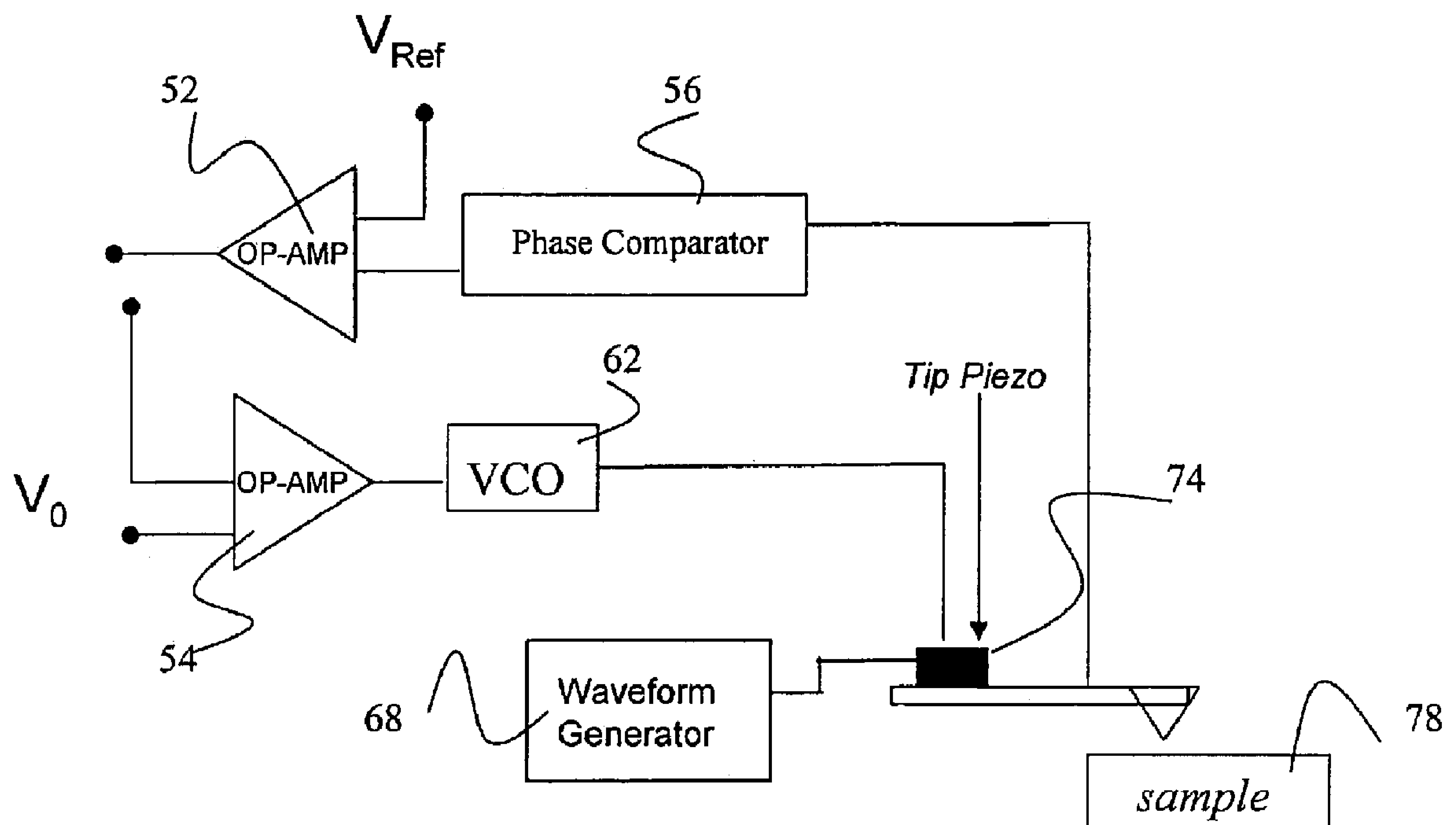


Figure 7. Micro-cantilever resonance feedback circuit.

Fig. 8

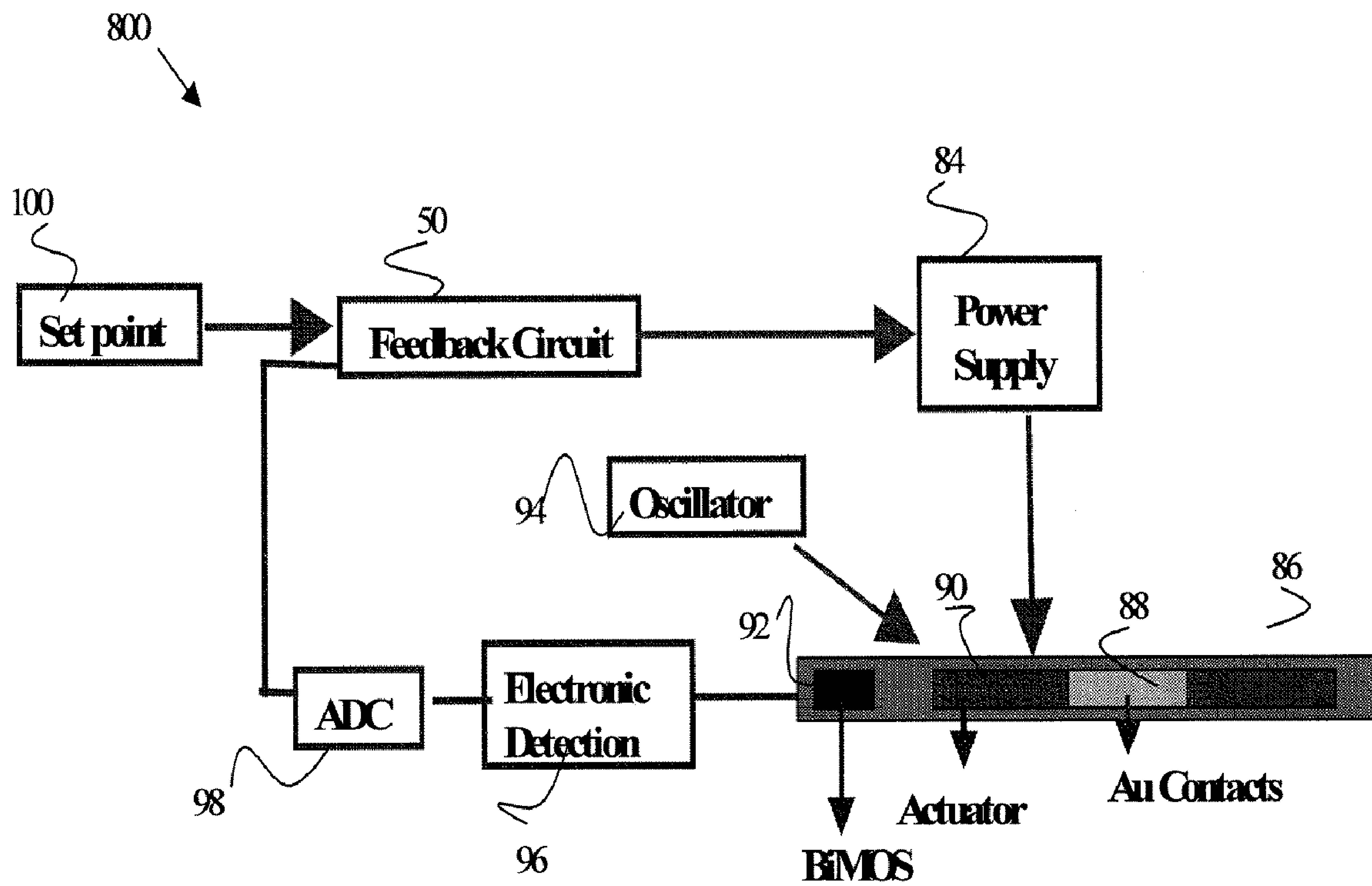
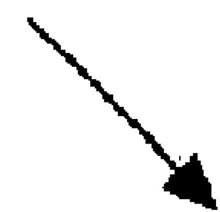


FIG. 9

900



Position a sample with respect to a cantilever. — 910

Vibrate cantilever tip at a first frequency. — 920

Vibrate sample at a second frequency. — 930

Detect interaction between the vibrating tip and the vibrating sample. — 940

Extract amplitude and phase information associated with surface and/or subsurface of the sample. — 950

Image surface and/or subsurface characteristics using the amplitude and phase information. — 960

Maintain tip and sample vibration at resonance frequencies. — 970

SCANNING NEAR FIELD ULTRASOUND HOLOGRAPHY

CROSS-REFERENCE TO RELATED APPLICATIONS

This application is a continuation of non-provisional application Ser. No. 11/244,747 filed Oct. 6, 2005, which is a continuation-in-part of non-provisional application Ser. No. 10/913,086 filed Aug. 6, 2004, which claims priority to provisional application Ser. No. 60/494,532 filed Aug. 12, 2003, which are hereby incorporated herein by reference in their entirety.

STATEMENT REGARDING FEDERALLY SPONSORED RESEARCH OR DEVELOPMENT

N/A

BACKGROUND OF THE INVENTION

Known acoustic microscopes are used for imaging structures such as integrated circuit (IC) structures. The spatial resolution, w, of an acoustic microscope is given by:

$$w = 0.51\frac{\vartheta}{f \cdot NA}$$

where ∂ is the speed of sound in the coupling medium, f is the frequency of the acoustic/ultrasonic wave, and N.A. is the numerical aperture of the lens. For a frequency of 1 GHz, the nominal spatial resolution attainable is approximately 1.5 μm. Further, the acoustic microscope has two other major roadblocks in getting high resolution: (1) impedance mismatches and coupling fluid attenuation that is proportional to f. Higher resolution alternatives for nondestructive mechanical imaging include the atomic force microscope (AFM) or scanning probe microscope (SPM) platforms. A few examples include: force modulation microscopy (FMM) as described by P. Maivald, H. J. Butt, S. A. C. Gould, C. B. Prater, B. Drake, J. A. Gurley, V. B. Elings, and P. K. Hansma in *Nanotechnology* 2, 103 (1991); ultrasonic-AFM as described by U. Rabe and W. Arnold in *Appl. Phys. Lett.* 64, 1423 (1994); and ultrasonic force microscopy (UFM) as described by O. V. Kolosov, K. Yamanaka in *Jpn. J Appl. Phys.* 32, 1095 (1993); by G. S. Shekhawat, O. V. Kolosov, G. A. D. Briggs, E. O. Shaffer, S. Martin and R. Geer in Nanoscale Elastic Imaging of Aluminum/Low-k Dielectric Interconnect Structures, presented at the *Material Research Society, Symposium D*, April 2000 and published in *Materials Research Society Symposium Proceedings, Vol* 612 (2001) pp. 1; by G. S. Shekhawat, G. A. D. Briggs, O. V. Kolosov, and R. E. Geer in Nanoscale elastic imaging and mechanical modulus measurements of aluminum/low-k dielectric interconnect structures, *Proceedings of the International Conference on Characterization and Metrology for ULSI Technology, AIP Conference Proceedings*. (2001) pp. 449; by G. S. Shekhawat, O. V. Kolosov, G. A. D. Briggs, E. O. Shaffer, S. J. Martin, R. E. Geer in *Proceedings of the IEEE International Interconnect Technology Conference,* 96-98, 2000; by K. Yamanaka and H. Ogiao in Applied Physics Letters 64 (2), 1994; by K. Yamanaka, Y. Maruyama, T. Tsuji in Applied Physics Letters 78 (13), 2001; and by K. B. Crozier, G. G. Yaralioglu, F. L. Degertekin, J. D. Adams, S. C. Minne, and C. F. Quate in Applied Physics Letters 76 (14), 2000. Each of these techniques is traditionally sensitive to the static elastic properties of the sample surface.

Recent developments in atomic force microscopes have involved the application of ultrasonic frequency (MHz) vibrations to the sample under study and non-linearly detecting of the deflection amplitude of the tip at the same high frequencies. With this arrangement, which is commonly identified as an ultrasonic force microscope, the ultrasonic frequencies employed are much higher than the resonant frequency of the microscope cantilever. The microscope exploits the strongly non-linear dependence of the atomic force on the distance between the tip and the sample surface. Due to this non-linearity, when the surface of the sample is excited by an ultrasonic wave, the contact between the tip and the surface rectifies the ultrasonic vibration, with the cantilever on which the tip is mounted being dynamically rigid to the ultrasonic vibration. The ultrasonic force microscope enables the imaging and mapping of the dynamic surface viscoelastic properties of a sample and hence elastic and adhesion phenomenon as well as local material composition which otherwise would not be visible using standard techniques at nanoscale resolution.

The drawback of ultrasonic microscopy is that it measures only the amplitude due to ultrasonically induced cantilever vibrations. Moreover, where the sample is particularly thick and has a very irregular surface or high ultrasonic attenuation, only low surface vibration amplitude may be generated. In such circumstances the amplitude of vibration may be below the sensitivity threshold of the microscope in which case measurement is impossible. Moreover, none of the above mentioned techniques measures with high resolution the acoustic phase, which is very sensitive to subsurface elastic imaging and deep defects identification which are lying underneath the surface, without doing any cross sectioning of the samples.

Out-of-plane vibrations created by non-linear tip sample interaction make a very hard elastic contact with the sample surface. Ultrasonic force microscopy (UFM) uses the same method except for a amplitude component rather than a phase contrast. If non-linearity is present in the system, most of the phase contrast will come from the surface and not from a surface/sub-surface phase contrast. Additionally, non-linear tip sample interaction may not provide results for soft materials. Furthermore, in UFM, high mechanical contrast may be acquired with little sub-surface contrast.

BRIEF SUMMARY OF THE INVENTION

The present invention relates to a high spatial resolution phase-sensitive technique, which employs a scanning near field ultrasonic holography methodology for imaging buried or other subsurface structures or variation in the specimen. Scanning near field ultrasound holography (SNFUH) uses a near-field approach to measure time-resolved variations in ultrasonic oscillations at a sample surface. As such, it overcomes the spatial resolution limitations of conventional phase-resolved acoustic microscopy (i.e. holography) by eliminating the need for far-field acoustic lenses.

The fundamental static and dynamic nanomechanical imaging modes for the instrument of the present invention are based on nanoscale viscoelastic surface and subsurface (e.g., buried nanostructure) imaging using two-frequency ultrasonic holography. The scanning near-field ultrasonic technique of the present invention vibrates both the cantilevered tip and the sample at ultrasonic/microwave frequencies. The contact, soft-contact and near-contact modes of tip-sample

interaction enable the extraction of the surface acoustic waves signal between the two ultrasonic vibrations.

Perturbations to the phase and amplitude of the surface standing acoustic wave may be locally monitored by the SPM acoustic antenna via lock-in and SNFUH electronic module. As the specimen acoustic wave gets perturbed by buried features, the resultant alteration in the surface acoustic standing wave, especially its phase, is effectively monitored by the SPM cantilever. Thus, within the near-field regime (which enjoys superb spatial resolution), the acoustic wave (which is non-destructive and sensitive to mechanical/elastic variation along its path) is fully analyzed, point-by-point, by the SPM acoustic antenna in terms of its phase and amplitude. Thus, as the specimen is scanned across, a pictorial representation of specimen acoustic wave's perturbation is recorded and displayed, to offer quantitative account of the internal features of the specimen.

Certain embodiments provide contact, soft (e.g., intermittent) contact, and/or near contact modes of operation to identify surface and subsurface (e.g., buried) characteristics of a specimen. Additionally, an SNFUH electronic module extracts surface acoustic phase and amplitude with or without non-linear tip sample interaction

These and other advantages and novel features of the present invention, as well as details of an illustrated embodiment thereof, will be more fully understood from the following description and drawings.

BRIEF DESCRIPTION OF THE SEVERAL VIEWS OF THE DRAWING

FIG. **1** is a block diagram illustrating the scanning probe microscope with scanning near field ultrasound holography of the present invention;

FIG. **2** is an illustration of atomic force microscopy of the present invention with a vibrating cantilever tip and vibrating sample;

FIG. **3**(A) is a schematic illustration of a model nanoparticle system for validation of SNFUH.

FIG. **3**(B) shows an AFM (topography) image with a featureless top polymer surface.

FIG. **3**(C) shows the phase image of SNFUH revealing buried gold nanoparticles with high definition.

FIG. **4**(A) shows a schematic of a model test sample for detecting embedded defects/voiding in shallow trenches.

FIG. **4**(B) shows an AFM (topography) image with a uniform coating of dielectric material.

FIG. **4**(C) shows a phase image of SNFUH that reveals the surface elastic contrast and embedded voiding in polymer coating over nitride and hardening of the coating at the trench walls.

FIG. **4**(D) shows a line profile across a void marked across X-Y.

FIG. **5**(A) shows an AFM topography of malaria-infected red blood cells.

FIG. **5**(B) shows an SNFUH phase image from malaria-infected red blood cells.

FIG. **5**(C) represents an AFM topography of early-stage incubation of parasite infection in malaria-infected red blood cells.

FIG. **5**(D) represents an SNFUH phase image of early-stage incubation of parasite infection in malaria-infected red blood cells.

FIG. **6**(A) depicts AFM (topography) imaging of a copper-low K dielectric interconnect system.

FIG. **6**(B) depicts SNFUH imaging of a copper-low K dielectric interconnect system.

FIG. **6**(C) shows a line profile across the voids in FIG. **6**(B).

FIG. **7** shows a feedback control circuit used in accordance with an embodiment of the preset invention.

FIG. **8** illustrates the feedback circuit in the context of an embodiment of an electronic readout system used in accordance with an embodiment of the present invention.

FIG. **9** illustrates a flow diagram for a method for scanning near field acoustic imaging used in accordance with an embodiment of the present invention.

The foregoing summary, as well as the following detailed description of certain embodiments of the present invention, will be better understood when read in conjunction with the appended drawings. For the purpose of illustrating the invention, certain embodiments are shown in the drawings. It should be understood, however, that the present invention is not limited to the arrangements and instrumentality shown in the attached drawings.

DETAILED DESCRIPTION OF THE INVENTION

Certain embodiments of the present invention are directed to a nondestructive, high resolution, sub-surface nanomechanical imaging system. The system is capable of directly and quantitatively imaging the elastic (static) and viscoelastic (dynamic) response of a variety of nanoscale materials and device structures with spatial resolution of a few nanometers depending on the ultrasonic frequencies. For viscoelastic high resolution sub-surface nanomechanical imaging the target maximum probe frequency is around 5-10 GHz, for example. In an embodiment, the maximum relative phase resolution at this frequency is estimated to be 0.001° leading to a viscoelastic time resolution of less than <1 ps. The instrument of certain embodiments of the present invention operates in a manner similar to commercially available scanning probe microscopes (SPMs) in that quantitative, digital, rastered, nanometer-scale images are obtained of the sample elastic modulus, and sample viscoelastic response frequency. The instrument also provides conventional SPM imaging modes including topography, frictional, and force modulation imaging.

Applications for certain embodiments of the present invention are numerous and represent areas of critical need in Molecular electronics, Nanosystems (NEMS), and Nanotechnology, in general. By combining the nanometer-scale spatial resolution of conventional SPMs with the sub-surface defect identification and imaging capabilities of acoustic or ultrasonic microscopes, the instrument fills a critical need in characterizing and investigating the nanomechanics of nanoscale systems. The SNFUH system and method may be used for: (1) in-vitro imaging of biological specimens, tissues and cells, (2) nanomechanical imaging of buried structures, inclusions in nanocomposites, failure analysis in IC structures and devices, (3) mechanical properties of low-K materials, (4) stress variation in 3D structures and interconnects, (5) flaw imaging in ceramics and quantitative evaluation of mechanical properties, etc.

Certain embodiments of the present invention are based on Nanoscale viscoelastic surface and subsurface (e.g., buried nanostructure) imaging using two-frequency ultrasonic holography. This is essentially a 'scanning near-field' ultrasound technique, where both the cantilevered tip **10** and the sample **12** are vibrated at ultrasonic/microwave frequencies. Contact and soft-contact tip-sample interaction enables the extraction of surface acoustic wave amplitude and phase with high resolution.

In SNFUH mode, perturbation to the standing surface acoustic wave resulting from specimen acoustic wave scat-

tering is monitored by an SPM acoustic antenna. The resulting cantilever deflection merely follows the perturbation to the surface standing acoustic wave, which represents the dissipative lag/lead in the surface response with respect to the tip reference frequency (i.e. the time of flight delay of the specimen acoustic waves reaching the sample surface). Extracting the spatial dependence of this phase term provides image contrast indicative of the relative elastic response of the buried structures, interfaces, and embedded defects to the specimen acoustic wave and thus the resultant perturbation to the standing surface acoustic wave.

Certain embodiments of the present invention provide a system that measures subsurface (e.g., buried) defects, delaminations; cracks; stress migration and etc., while maintaining the high resolution of the atomic force microscope. It utilizes (1) an atomic force microscopy system having a cantilever **14** with a tip **10** at a free end sitting on top of the vibrating device **16** for supplying vibrations to the cantilever at a frequency greater than cantilever resonance frequency, (2) a sample **12** having a vibration device **18** sitting under it for providing high frequency excitations and (3) an optical detector or other detector for detecting movement of the cantilever. It detects the beats, products, additions frequencies and beats, products of their harmonics and modulated waveforms, when the vibrating tip interacts with the vibrating sample, which falls within its detection range. With this embodiment, it is possible to recover surface acoustic wave phase information of the tip-surface mechanical interaction, which allows measurement of viscoelastic properties and enables the application of acoustic holography algorithms for imaging nanoscale sized sub-surface (e.g., buried) defects. The microscopy apparatus utilizes scanning Near Field Ultrasound Holography (e.g., SNFUH) for high resolution nanomechanical imaging of buried defects and structures.

The surface acoustic wave's amplitude and phase are experimentally extracted from the tip deflection signal via lock-in detection. The phase sensitivity of this measurement is involved in extracting time-resolved mechanical properties of materials as well as potentially enabling subsurface imaging (e.g., buried nanostructures).

Certain embodiments of the present invention detect the phase of transmitted acoustic wave directly at wafer/device surface. Further, certain embodiments of the present invention detect the phase of surface acoustic wave directly at wafer/device surface. Further, certain embodiments of the present invention utilize scanning nanoprobe phase detection so as to eliminate the need for acoustic lenses. The Nanoprobe Acoustic Antenna (AFM Tip) of certain embodiments of the present invention is advantageous because, for example, it provides induction of MHz-GHz nanoprobe mechanical oscillations via high frequency flexural mode excitation, i.e. the mechanical wave guide and the cantilever monitors the phase shifts between tip **10** and sample **12** acoustic/ultrasonic vibrations.

As shown in FIGS. **1** and **2**, two oscillations are applied to the tip **10** and sample **12** by two matched piezo crystals **16** and **18** attached to the Si substrate of the tip and the base of the sample, respectively. Each piezo **16**, **18** is driven by a separate waveform with a SNFUH electronic module **36** providing the input frequency to an RF lockin amplifier **40** for surface acoustic wave (SAW) amplitude and phase extraction. The SNFUH electronic module **36** selects beats, product, and/or addition frequencies for example, to aid in performing holography in contact, soft-contact, and near-contact modes. Additionally, the SNFUH electronic module **36** allows SNFUH to be performed in the linear regime of tip-sample interaction. In an embodiment, the SNFUH electronic module **36** includes a mixer circuit, variable resistor(s), op-amp(s), band pass filter(s), and/or other filters for mixing frequency signals and selecting frequency products, additions and beats, for example.

Any Scanning Probe Microscope (SPM) may serve as the base platform. A signal access module (SAM) **22** is used as the input site for SNFUH, and modulus-calibration signals. The integrated piezo (for high frequency excitation) will enable ultrasonic excitation of higher-order flexural resonances of the cantilever tip **10** to provide the ultrasonic vibration.

The sample ultrasonic vibration is driven by function generator **32**. Second function generator **34** applies the sample ultrasonic vibration. The resulting differential output signal from detector is accessed with the signal access module (SAM) **22** and acts as the input to RF lockin amplifier **30** or similar lockin amplifier for extraction of SAW amplitude and phase. The lockin response signal constitutes a SAW amplitude and phase which act as a input into the signal acquisition electronics **46**, via the SAM **22**, for image display and analysis. The SNFUH electronic module circuit **36** extracts beats, products of fundamental and harmonics, and/or modulated waveform(s), which serve as a reference for a RF lockin amplifier **40** or other lockin amplifier, for example. The differential output of optical detector (A-B) is input, via the SAM **22**, into the RF lockin **40**. The resulting output constitutes the SNFUH image signal. A computer **44** or other processor running data acquisition/analysis software, such as Lab View or other data acquisition and/or analysis software, acquires both the A-B signal from the digital scope and the lockin. In an embodiment, a switch may be included to select an SNFUH or UFM signal for acquisition, for example.

In an embodiment, the sample piezo consists of an insulator/electrode/piezo/electrode/insulator blanket multilayer (e.g., 10 cm×10 cm) stack. The insulators consist of epoxied machinable ceramics or thin, spin-cast polymer coatings, dependent upon ultrasonic coupling efficiency. The Cr/Au electrodes or other similar electrodes provide electrical contact between the piezo and the second function generator **34**. The assembly is counter-sunk into a modified SPM sample mount.

As shown in FIG. **2**, using SNFUH, a high frequency acoustic wave is launched from below the specimen **12**, while another high frequency acoustic wave is launched at least a slightly different frequency is launched on the SPM cantilever **10**. The SNFUH electronic module **36** is used to spatially monitor the phase perturbation to a standing surface acoustic wave, which results from a scattered specimen acoustic wave. The resonant frequency of the cantilever, f_0, may be in the 10-100 kHz range, for example.

Certain embodiments may also include a feedback circuit, such as the cantilever resonance feedback circuit **50** depicted in FIG. **7**. The feedback circuit **50** includes a first op amp (OA) **52**, a second op amp (OA) **54**, a phase compensator (PC) **56**, a voltage-controlled oscillator (VCO) **62**, a waveform or function generator **68** and a cantilever tip having a piezo transducer **74** interacting with a sample **78**.

For SNFUH operation to be uniquely calibrated across samples, phase of the cantilever may be fixed. In order to fix the phase of the tip, a resonance feedback circuit, said as feedback circuit **50** may be employed. The feedback circuit **50** maintains the tip carrier frequency at resonance and fixes or sets the phase, so that the tip phase is a stable reference for sample phase. For example, at higher frequencies (e.g., 150 MHz-10 GHz), the cantilever easily strays from its resonance, and feedback may be used to maintain the cantilevered tip frequency at resonance. Both the sample and cantilever may

maintain their resonance frequencies to generate a high resolution viscoelastic response. In an embodiment, the system may operate in feedback mode with the feedback circuit **50** generating feedback for frequency resonance, or the system may operate without the feedback circuit **50** activated.

In the feedback circuit **50**, the voltage-controlled oscillator **62** drives the tip piezo transducer **74**. The VCO **62** is connected through the phase compensator **56**, which acts as an input to an op amp pair **52**, **54** for feedback control. If the cantilever resonance frequency shifts during scanning, the reduction in tip vibration amplitude will reduce the voltage across the piezo transducer on the cantilever. This voltage will cause a shift in the PC **56** output. The shift in PC output will bring the VCO **62** back into resonance.

FIG. **8** illustrates the feedback circuit **50** in the context of an embodiment of an electronic readout system **800** used in accordance with an embodiment of the present invention. The electronic readout system **800** may be a MOSFET embedded electronic readout, for example. Using embedded MOSFET as electronics feedback may provide a current sensitivity of $\Delta I_d/I_d=10^{-6}$/nm of cantilever bending. Deflection sensitivity of the electronic readout may be of the same order as optical feedback detection, for example. In an embodiment, deflection sensitivity may be approximately three orders of magnitude higher than existing passive and active detection technologies, such as piezoresistive detection. In an embodiment, a high signal-to-noise ratio and minimal 1/f noise allow the MOSFET embedded electronic readout to be used for electronic feedback in SPM's (scanning probe microscopes), for example.

The feedback circuit **50** may be used to control a power supply **84** which supplies power to a piezo **86**. The piezo **86** includes contacts, such as Au (gold) contacts **88**, as well as an actuator **90** and a BiMOS transistor **92**. The piezo **86** is driven by an oscillator **94**. Feedback from the oscillating piezo **86** is gathered by the electronic detection unit **96**. The feedback signal from the electronic detection unit **96** is converted using the analog to digital converter (ADC) **98** and fed into the feedback circuit **50** for control of the power supply **84**. Set point **100** provides a basis or reference for operation of the feedback circuit **50**. Feedback from the circuit **50** helps to ensure that the tip and the sample are being vibrated at their respective resonance frequencies, for example.

An example of viscoelastic nanomechanical imaging is shown in FIG. **3**. FIG. **3**(A) shows gold nanoparticles dispersed on a polymer coated substrate are buried under an approximately 500 nm thick polymer layer. Use of a model polymer-nanoparticle composite demonstrates the high lateral spatial resolution and depth sensitivity of the SNFUH approach. A specimen consisting of gold nanoparticles buried deep underneath a polymer cover layer was prepared by dispersing colloidal gold nanoparticles on a polymer (poly (2-vinylpyridine)-PVP) coated silicon substrate. The gold nanoparticles have an average diameter of 15 nm and are well dispersed on the film surface. The nanoparticles were then fully covered with another polymer film about 500 nm thick, as shown schematically in FIG. **3**(A). The normal AFM topography scan, FIG. **3**(B), shows a smooth featureless surface of top polymeric layer with surface roughness of approximately 0.5 nm. On the other hand, the phase image of SNFUH, FIG. **3**(C), shows well-dispersed gold nanoparticles buried approximately 500 nm deep from the top surface. The contrast in the phase image of SNFUH arises from the elastic modulus difference between the polymer and gold nanoparticle, which induces the time dependent phase delay of the acoustic waves reaching the sample surface.

To demonstrate the efficacy of SNFUH in identifying underlying defects in narrower trenches, shallow trench structures may be fabricated as shown in FIG. **4**(A). The trenches are etched in SOD (spin-on-dielectric) with a 50 nm thin layer of LPCVD Si_3N_4 as a capping layer and then Si_3N_4 is etched down in the 1 μm deep trenches using the wet processing. Trench width in the example is about 400 nm. A 500 nm thick layer of polymer [Benzocyclobutene (BCB)] was spin-coated followed by thermal annealing for curing the polymer.

FIG. **4**(A) shows a schematic of series of isolated shallow trench structures. FIG. **4**(B) shows a conventional AFM topography image, while FIG. **4**(C) is a corresponding (simultaneously recorded) SNFUH phase image. The typical $7.5\times7.5\ \mu m^2$ topography scan shows uniform and contiguous polymeric coating on SiN and inside the trenches. On the other hand, the corresponding SNFUH phase image shown in FIG. **4**(B) reveals phase contrast reminiscent of embedded voiding within the polymer, and at the SiN-polymer interfaces. The dark contrast in the phase image in polymer coated SiN lines corresponds to voids at polymer-SiN interface, i.e. voiding underneath the contact. The contrast is due to the distinct viscoelastic response from the specimen acoustic wave from the voids, for example. A hardening of the polymer in the trench and its sidewall is also evident in the phase image, which results from thermal annealing and possibly poor adhesion with SOD, for example. FIG. **4**(D) shows a line profile of phase across X-Y from FIG. **4**(C). A subsurface phase resolution of 50 mdeg may be achieved, for example. Current methods of diagnosis employ destructive approaches such as wet etching followed by SEM imaging, which are undesirable. Thus, SNFUH may be an improved tool-set for such subsurface metrology needs.

The efficacy of SNFUH to imaging embedded or buried sub-structures in biology is demonstrated in FIG. **5**, which depicts high resolution and remarkably high contrast arising from malaria parasites inside infected red blood cells (RBCs). FIG. **5** demonstrates early stage direct and real-space in-vitro imaging of the presence of parasites inside RBCs without labels or sectioning of cells, and under physiologically viable conditions. Plasmodium falciparum strain 3D7 was cultured in-vitro by a modification of the method of Haldar et al. Parasites were synchronized to within 4 hours using a combination of Percoll purification and sorbitol treatments, cultured to 10% parasitemia, and harvested at the indicated times, for example.

SNFUH imaging may be performed using the near-contact mode method for imaging soft structures, for example. An SNFUH electronic module may be used to bring the cantilever in near-contact mode and then the sample was subsequently scanned over the RBCs while maintaining the near-field regime. FIGS. **5**(A) and **5**(B) show AFM topography images and SNFUH phase images from infected RBCs, respectively. The AFM topography image shows the typical surface morphology of infected RBC, while the SNFUH phase image shows high contrast from the parasite residing well inside the RBC. In addition to several other features reminiscent of membrane proteins and sub-cellular contents, multiple parasites are clearly evident. In order to further demonstrate the capability of SNFUH for early stage diagnosis of parasite infection, RBCs incubated for only four hours are examined, which is difficult to validate by other non-invasive technique (e.g., fluorescence tagging). FIGS. **5**(C) and **5**(D) show a pair of images similar to those in FIGS. **5**(A) and **5**(B). SNFUH may be sensitive to early stage parasite infection in RBC, as reflected by image contrast consistent with parasite infection, for example.

FIG. **6** shows a series of low-K dielectric polymer and copper lines with lateral dimension of about 200 nm for the polymer and around 60 nm for copper. FIG. **6**(A) shows the conventional topography image, while FIG. **6**(B) is the corresponding (simultaneously recorded) SNFUH phase image. The typical 1400×1400 nm^2 topography scan shows uniform and contiguous polymer and copper lines. However, the corresponding SNFUH phase image shown in FIG. **6**(B) reveals phase contrast reminiscent of sub-surface voiding in copper lines. FIG. **6**(C) shows a line profile across the voids. The dark contrast in the phase image of copper lines corresponds to voids underneath the metal. The presence of this contrast in phase image implies that there is insufficient metal filling at the bottom, i.e. voiding underneath the contact, which undergoes a distinct viscoelastic response. Interestingly, a hardening of the polymeric regions and its sidewall is also evident in the phase image, which results from RIE processing and chemical-mechanical-polishing (CMP). SNFUH may serve as a tool-set for such sub-surface metrology challenges.

Thus, SNFUH may be used to facilitate: (1) quantitative high resolution nanomechanical mapping of subsurface (e.g., buried) structures to identify process-induced mechanical variations and/or nanoscale cohesive defects; (2) nanomechanical viscoelastic (dynamic) imaging to specifically investigate surface and subsurface interfacial adhesive (bonding) response, etc.

Other applications for the system and method of the present invention include: (1) non-destructive imaging of subsurface defects in 3D interconnects and stress migration along the devices due to electrical biasing; (2) non-destructive inspection for interconnect nanotechnology for nanometer-scale resolution, to enable imaging of electro-mechanical defects (e.g. nanotube contacts) and to enable imaging of nanoscale integrity in molecular interconnect assemblies; (3) subsurface nano-cracks, stress, delamination identification in ferroelectrics, ceramics and micromechanical structures and devices; (4) non-destructive defect review and process control in integrated IC materials and devices to provide modulus measurement for soft materials (i.e. porous dielectrics) and to provide void and delamination defect detection to avoid off-line, cross-sectional failure analysis; (5) self assembled monolayers and subsurface defects in biological cells and materials, in-vitro imaging of biological cells, tissues and membranes, nano-bio mechanics and (6) quantitative extraction of elastic modulus with high accuracy.

FIG. **9** illustrates a flow diagram for a method **900** for scanning near field holography imaging used in accordance with an embodiment of the present invention. First, at step **910**, a sample is positioned with respect to a cantilever for nanomechanical imaging of the sample. Surface and/or subsurface imaging may be performed with respect to the sample, such as a tissue or other sample. At step **920**, a tip of the cantilever is vibrated at a first frequency. The frequency may be a first microwave, ultrasonic, or other acoustic frequency, for example. Then, at step **930**, the sample is vibrated at a second frequency, such as a second microwave, ultrasonic, or acoustic frequency, for example. In an embodiment, the second frequency vibrates the sample at a frequency that is offset from the first frequency vibrating the tip. In an embodiment, the tip and sample piezos are vibrated at their respective resonance frequencies.

Next, at step **940**, interaction between the vibrating tip and the vibrating sample is detected. The interaction may be a physical interaction and/or a non-contact signal interaction between the tip and sample, for example. The interaction may constitute movement of the tip, for example. Tip movement may be provided as a tip deflection signal, for example. In an embodiment, the interaction may include a linear and/or non-linear interaction between the tip and the sample. In an embodiment, SNFUH may be performed using the linear tip-sample interaction in soft contact and near contact mode to obtain the high resolution sub-surface phase. SNFUH may be performed in soft and near contact modes to obtain the sub-surface information, such as buried defects or variations.

At step **950**, amplitude and phase information associated with the surface acoustic waves of the sample are extracted. Amplitude and phase information may be extracted from the tip deflection signal using lock in detection, for example. In an embodiment, subsurface mechanical data, such as interfacial bonding, regarding the sample may also be extracted from the tip deflection signal.

At step **960**, surface and/or subsurface characteristics of the sample may be imaged using the amplitude and phase information. In an embodiment, a spatial variation of surface and subsurface viscoelastic phase may be imaged, for example. In an embodiment, a characteristic viscoelastic response time of the sample may be defined based on the amplitude and phase information. Then, at step **970**, vibration of the cantilever tip is maintained at a tip piezo resonance frequency, and vibration of the sample is maintained at a sample resonance frequency. Feedback, such as electrical feedback, may be provided to maintain the tip resonance frequency and the sample resonance frequency.

In an embodiment, product frequencies may be used with optical detection to obtain biological imaging with high subsurface resolution. The sample and cantilever are excited at their fundamental resonance frequencies (e.g., 1.96 MHz and 3.28 MHz, respectively). Additionally, the individual sample and cantilever carriers are modulated with one or more modulation frequencies (e.g., 25 kHz and 35 kHz, respectively). Next, a combination of a SNFUH electronic module and RF lock in band pass filter outputs a product of the two modulated waveforms. The product output is then fed into a RF lock in amplifier reference input.

Using product frequencies allows improved selection of carrier frequencies. In an embodiment, the larger the frequency of acoustic oscillations, the higher the order of phase contrast that may be obtained from SNFUH images. Thus, smaller features not seen at lower carrier frequencies may be detected using a higher frequency carrier. Additionally, use of product frequencies allows use of non-matching tip and cantilever piezos.

In an embodiment, forces between the cantilever and sample may be controlled during SNFUH operation in near contact mode. Contacting the cantilever with the biological samples may rupture the samples. However, near contact operation allows monitoring and subsurface imaging of soft structures. Near contact mode operation may provide subsurface imaging of soft structures as well as providing quantitative analysis of biological structures, cells and/or tissues, for example.

In an embodiment, beat frequencies may be used to monitor samples in near contact mode. Alternatively, frequency addition may be used for sample monitoring in near contact mode. In an embodiment, harmonics, as well as or in addition to fundamental frequencies, may be used in beat frequency, product frequency, and/or frequency addition (sum) operation. For example, the system may perform optical and/or electronic detection according to a variety of frequency strategies up to 1000 MHz with a very thin film of ZnO.

In an embodiment, cantilever and sample carrier frequencies may be modulated with amplitude modulation. For example, two carriers, a carrier for the cantilever and a carrier for the sample are amplitude modulated individually. In this

configuration, the tip-sample assembly may be excited with a higher frequency (with or without matching piezos). Then, an amplitude modulated waveform may be obtained from both the cantilever and sample and input to a SNFUH electronic module. The output of the electronic module is a product/difference/addition frequency. A beat or difference frequency is a difference between modulation frequencies, for example.

In an embodiment, an electronic readout device may be implemented with the sample monitoring system. An example of such a readout device may be the readout device described in U.S. patent application Ser. No. 10/996,274, filed on Nov. 23, 2004, entitled "Method and System for Electronic Detection of Mechanical Perturbations Using BiMOS Readouts", which is herein incorporated by reference. In an embodiment, using a readout circuit allows product frequencies to be used without modulation since operation is not limited by response time as with optical photodiodes, for example.

In an embodiment, use of electronic detection eliminates optical detection of amplitude and phase removes or eases limitations imposed by a photo-detector response frequency, such as a 1 MHz photo-detector response frequency. Electronic detection aids in fabricating multi-active probes with on-chip integrated piezo-actuator (e.g., ZnO) and embedded MOSFET feedback electronics. Additionally, electronic detection does not limit detection of subsurface features based on beat frequency. Multiples of frequencies may be used to enhance both amplitude and subsurface phase contrast, and thus the viscoelastic response. Enhanced viscoelastic response results in enhanced phase contrast from features less than 50 nm, for example, which may be difficult to detect using only beat frequencies.

Thus, certain embodiments provide a scanning near field ultrasound holography (SNFUH) technique to image high resolution buried nanostructures, defects, 3D tomography, identification of individual layers in multilayer thin film stacks and dopant mapping, for example. Certain embodiments integrate three approaches: a combination of scanning probe microscope platform (which enjoys excellent lateral and vertical resolution) coupled to micro-scale ultrasound source and detection (which facilitates "looking" deeper into structures, section-by-section) and a holography approach (to enhance phase resolution and phase coupling in imaging). Certain embodiments provide near field, ultrasonic holography, near field microwave holography, or other near field acoustic holography for surface and subsurface imaging in nano- and micro-specimens, such as biological, mechanical, and electrical specimens. Certain embodiments allow SNFUH imaging using linear and/or non-linear interactions between cantilever and specimen in contact, soft contact and/or near contact mode, for example.

As a result, the technique allows subsurface flaw imaging in nano- and micro-composites, MEMS, CMOS, and heterostructures, for example. The technique also provides in-vitro imaging of biopolymer, biomaterials and biological structures (e.g. viewing cell-membrane or implant-bio interface). Additionally, certain embodiments detect voiding and subsurface defects in low-K dielectric materials and interconnects, as well as stress migration and defect analysis in 3D interconnects and MEMS. Certain embodiments facilitate dopant profiling and modulus mapping in non-contact mode and also provide non-invasive monitoring of molecular markers/tags-signal pathways, for example.

In an embodiment, a high frequency (e.g., on the order of hundreds of MHz) acoustic wave is launched from the bottom of the specimen, while another wave is launched on the AFM cantilever. These acoustic waves are mixed together through a SNFUH electronic module, which includes a combination of various filters, mixers, feedback electronics and electronic components used to obtain a desired product and addition of fundamental resonances and related harmonics (in addition to difference frequencies). The resulting mixed wave is monitored by the AFM tip, which itself acts as an antenna for both phase and amplitude. As the specimen acoustic wave gets perturbed by buried features, especially its phase, the local surface acoustic waves are very effectively monitored by the AFM tip. Thus, within the near-field regime (which enjoys superb lateral and vertical resolution), the acoustic wave (which is non-destructive and sensitive to mechanical/elastic variation in its "path") is fully analyzed, point-by-point, by the AFM acoustic antenna in terms of phase and amplitude. Thus, as the specimen is scanned across, a pictorial representation of acoustic wave's perturbation is fully recorded and displayed, to offer a "quantitative" account of internal microstructure of the specimen.

The SNFUH system is operational in the linear and near-contact regime of tip-sample interaction and proves effective for in-vitro imaging of biological cells and tissues using the SNFUH electronic module, for example.

Thus, certain embodiments provide an electronic readout based on an embedded MOSFET to detect product frequencies, which is thereby not limited by an optical detector. Moreover, electronic readout may help in building a parallel SNFUH system for industrial application. In addition, a Brillion Zone Scattering technique may be used to map the modules of any surface in non-destructive way with greater efficiency than other methods.

Certain embodiments may be applied to microelectronics, especially as an advanced nanoscale surface and sub-surface metrology tool-set. Further, certain embodiments provide imaging for Nanoelectronics, reliability and failure analysis in Microsystems (MEMS), and Nanotechnology, in general, and especially biomolecular interconnects and BioMEMS. Additionally, certain embodiments provide in-vitro imaging of biological structures without having to "open-up" internal structures. By combining the nanometer-scale spatial resolution of conventional SPMs with the sub-surface imaging capabilities, certain embodiments may characterize the surface defects and structures with high resolution and will have further potential for developing nanoscale non-invasive 3D tomography, for example.

Scanning Near Field Ultrasound Holography (SNFUH) may be used, for example, in near contact and contact mode with product frequencies for the following structures and devices: (1) Investigating mechanical uniformity and process-induced mechanical modification of materials in integrated circuit (IC) structures and MEMS; (2) Real-time in-vitro biological imaging of red blood cells infected with malaria parasites; (3) Voiding in copper interconnects and (4) Non-invasive monitoring of nanoparticles buried under polymeric films. Such capabilities may complement cross-sectional imaging techniques such as SEM-EDS (scanning electron microscope-energy dispersive spectroscopy), TEM-EDS (transmission electron microscope-energy dispersive spectroscopy), TEM-EELS (transmission electron microscope-electron energy-loss microscopy), and ex situ STM (scanning tunneling microscopy) to investigate the nanomechanics and subsurface imaging of material interfaces, the uniformity of conformally deposited coatings, and mechanical defects in multilayer structures, for example.

Many other applications of the present invention as well as modifications and variations are possible in light of the above teachings. While the invention has been described with reference to certain embodiments, it will be understood by those

skilled in the art that various changes may be made and equivalents may be substituted without departing from the scope of the invention. In addition, many modifications may be made to adapt a particular situation or material to the teachings of the invention without departing from its scope. Therefore, it is intended that the invention not be limited to the particular embodiment disclosed, but that the invention will include all embodiments falling within the scope of the appended claims.

The invention claimed is:

1. A scanning near field ultrasound holography method for surface and subsurface imaging comprising:
- vibrating a cantilevered tip at a first ultrasonic frequency;
- vibrating a sample at a second ultrasonic frequency;
- detecting movement of the cantilevered tip interacting with the vibrating sample to provide a tip deflection signal;
- extracting surface acoustic wave (SAW) amplitude and phase information associated with the surface and subsurface of the sample using lock in detection; and
- using near field holography to enhance phase resolution and phase coupling in imaging.

2. The method of claim **1**, wherein said extracting step further comprises extracting surface acoustic wave (SAW) amplitude and phase information associated with the surface and subsurface of the sample using lock in detection and a scanning near field ultrasound holography (SNFUH) electronic module.

3. The method of claim **1**, wherein said second ultrasonic frequency vibrates said sample at a frequency offset from said first ultrasonic frequency.

4. The method of claim **1**, wherein said extracting step further includes extracting subsurface mechanical data regarding the sample from the tip deflection signal.

5. The method of claim **4**, wherein said subsurface mechanical data includes interfacial bonding.

6. The method of claim **1**, further comprising imaging a spatial variation of surface and subsurface viscoelastic phase.

7. The method of claim **1**, further comprising defining a characteristic viscoelastic response time of the sample based on said amplitude and phase information.

8. The method of claim **1**, further comprising maintaining said vibration of a cantilever tip piezo at a tip piezo resonance frequency and said vibration of said sample at a sample resonance frequency.

9. The method of claim **8**, further comprising providing electrical feedback to maintain said tip piezo resonance frequency and said sample resonance frequency.

10. The method of claim **1**, wherein frequencies are used to generate said tip deflection signal to monitor said sample in near contact mode.

11. The method of claim **1**, further comprising operating in near contact mode without contact between said cantilevered tip and said sample.

12. The method of claim **1**, further comprising:
- selecting, by a circuit, at least one of beat frequencies and product frequencies that serve as a reference for use with said lock in detection, said circuit being communicatively disposed between at least one function generator and a lockin amplifier.

13. The method of claim **1**, wherein said vibrating of said cantilevered tip is facilitated by a first piezo crystal attached to a substrate of said cantilevered tip, wherein said vibrating of said sample is facilitated by a second piezo crystal attached to a base of said sample, and wherein said first piezo crystal and said second piezo crystal are matched.

14. A scanning near field holography method for surface and subsurface imaging comprising:
- vibrating a cantilevered tip at a first microwave frequency;
- vibrating a sample at a second microwave frequency;
- detecting movement of the cantilevered tip interacting with the vibrating sample to provide a tip deflection signal;
- extracting surface acoustic wave (SAW) amplitude and phase information associated with the subsurface of the sample from the tip deflection signal using lock in detection; and
- using near field holography to enhance phase resolution and phase coupling in imaging.

15. The method of claim **14**, wherein said extracting step further comprises extracting SAW amplitude and phase information associated with the subsurface of the sample from the tip deflection signal using lock in detection and a scanning near field ultrasound holography (SNFUH) electronic module.

16. The method of claim **14**, further comprising:
- using a linear interaction between the cantilevered tip and the sample to detect beats at the surface of the sample; and
- extracting phase and amplitude signals for said first microwave frequency and said second microwave signal.

17. The method of claim **14**, further comprising imaging said surface and subsurface of said sample based on said amplitude and phase information of said tip deflection signal.

18. The method of claim **14**, further comprising:
- selecting, by a circuit, at least one of beat frequencies and product frequencies that serve as a reference for use with said lock in detection, said circuit being communicatively disposed between at least one function generator and a lockin amplifier.

19. An atomic force microscopy system, said system comprising:
- a cantilever, wherein said cantilever includes a tip at an end of said cantilever;
- a vibrating device configured to supply vibrations at a first frequency to said cantilever to generate vibration at said tip; and
- a detector configured to detect movement of said tip based on an atomic force between said tip and a surface of a sample, wherein said sample is vibrated at a second high frequency excitation different from said first frequency,
- wherein the atomic force microscopy system uses near field holography to enhance phase resolution and phase coupling in imaging.

20. The system of claim **19**, comprising:
- a function generator configured to generate a signal of at least one of said first microwave frequency and said second microwave frequency; and
- an electronic circuit configured to select at least one of beat frequencies and product frequencies that serve as a reference for use with said lock in detection, said electronic circuit being communicatively disposed between said function generator and a lockin amplifier.

* * * * *

UNITED STATES PATENT AND TRADEMARK OFFICE

CERTIFICATE OF CORRECTION

PATENT NO. : 7,798,001 B2
APPLICATION NO. : 12/244406
DATED : September 21, 2010
INVENTOR(S) : Gajendra Shekhawat et al.

It is certified that error appears in the above-identified patent and that said Letters Patent is hereby corrected as shown below:

Column 13, line 48 delete “wherein frequencies” and insert --wherein beat frequencies--.

Signed and Sealed this
Ninth Day of August, 2011

David J. Kappos
Director of the United States Patent and Trademark Office